Silicon Bonding Techniques for X-ray Optics

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A summary of R&D work carried out by the Experimental Facilities Division Optics Group (XFD-OP) through July 1995.

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Introduction

Some of the most efficient heat-exchanger designs for direct-cooled optics consist of two or more pieces of silicon single crystal bonded to each other and attached to a coolant manifold. Therefore, achieving successful silicon-to-silicon and silicon-to-metal bonds has become one of the goals of the high heat load (HHL) optics program. A viable bond for a cooled silicon optic has to satisfy the following requirements:

- strain free
- compatible with the coolant used
- radiation resistant
- coefficient of thermal expansion of the bonding agent must be close to that of silicon

The techniques that have been pursued by XFD-OP members are:

- Si-Si direct bonding
- Si-Si die attach paste bonding
- Si-Si and Si-metal epoxy bonding
- Si-Si and Si-metal glass frit bonding
- Si-metal gold-based solder

A description of each of these techniques and their performance follows. The work mentioned in this summary has been pursued by R.C. Blasdell, P.B. Fernandez, S. Felix Krasnicki, W.K. Lee, A.T. Macrander, and R.K. Smither.

Si-Si Direct Bonding

Direct bonding of silicon to silicon has been accomplished in the fabrication of x-ray analyzers for the milli-eV program.[1] However, achieving such bonds with minimum strain is still an open R&D program. For this application, a thin wafer (approximately 1 mm thick) is bonded onto a thicker piece (approximately 10 mm thick).

In principle, this bonding technique could be applied to thicker pieces of silicon (10-25 mm), thus making it useful for the fabrication of cooled optics.

The critical element in the process is the surface preparation. The mating surfaces have to be dust free, hydrophilic, and carefully dried, for example using a spinner. Room-temperature bonding occurs on contact; the strength of the bond is then increased by annealing at higher temperatures (200 to 800 °C). Details can be found in references 1 and 2.

The direct bonding technique has also been tested by joining two 4-inch diameter, 2-mm-thick silicon wafers.[3] The bond has been examined via x-ray diffraction, both over small areas using a double-crystal setup[4] and over the full face of the crystal assembly with our topography unit (see Appendix A for a brief description of the topography setup). The double-crystal data, which were taken using the Cu $K\alpha$ doublet, show a tendency to an overall bending of the two bonded wafers, but no local strains. These measurements were confirmed by the topographic data; Figure 1.a) shows the rocking curve when the full face of the crystal is illuminated by the x-ray beam. The width of the rocking curve is 4.7 arc seconds, which corresponds to a strain of 4.3 arc seconds. Figure 1.b) is a superposition of topographs taken at different points of the rocking curve, in 4 arc seconds steps. The bonded wafers show an overall convex shape, with a reasonably flat central region, shown as the wide reflecting band at the center of the crystal. These tests, and all the x-ray measurements later described in this report, were carried out using the $K\alpha 1$ line of copper at 8.05 keV.

The feasability of direct-bonding even thicker pieces of silicon has been demonstrated by Q.-Y. Tong and collaborators,[5] who have bonded two 4-inch diameter, 20-mm-thick pieces. The bonded pieces have not been examined using x-rays, so we do not have information on the strains that might have been induced by the bond.

The advantage of the direct bonding technique is the absence of any bonding agent. A potential disadvantage may be the involved preparation of the surface, in particular spinning large and odd-shaped pieces of silicon. We have procured a spinner, and we will assess the viability of the technique for fabricating HHL optics. The overall strain of the bonded parts may also be a source of concern, though strain may be less severe for thicker silicon pieces.

Si-Si Die Attach Paste Bonding

Silver-based die attach paste is a mixture of glass, silver, and organic compounds.[6] The silver in the glass paste serves several functions: it fills in any voids between the mating surfaces; it enhances the electrical and thermal conductivity of the bond; and it makes the coefficient of thermal expansion of the paste closer to that of silicon.

The surfaces to be bonded are lapped and cleaned. The crystals are weighed before and after assembling them with the die attach paste. Knowing the composition of the paste,[7] the weight loss required to get rid of 95% of the organic components in the paste is calculated. The assembly is then baked at low temperature (75 °C) until such weight loss is achieved; this process can take up to a day for large bonding areas. The bond is then cured by raising the temperature to 400-420 °C for 10-12 minutes. We have used this bonding procedure to fabricate five slotted, gallium-cooled silicon crystals. In each case, the slotted heat exchanger (top plate) was bonded to the silicon distribution plenum, and the assembly was then sealed to a metal manifold using rubber O-rings, see Figure 2.

Data taken using the APS/CHESS prototype undulator on one of the crystals assembled using die attach paste indicate that the bonding strains may be of the order of 5 arc seconds over an area of a few square millimeters.[8] A detailed study of the same crystal using the XFD-OP topography station shows a convex curvature of the diffraction planes near the surface. Figure 3.a) is the rocking curve obtained when the x-ray beam illuminates the whole 4-inch-diameter crystal, while Figure 3.b) is a topograph of the crystal, taken at the point where the intensity of the rocking curve is maximum. The width of the rocking curve is 3.9 arc seconds, which indicates a strain of 3.3 arc seconds, consistent with the undulator measurements. Figure 3.c) is a sketch of the slotted heat exchanger, which constitutes the top plate of the crystal assembly; the coolant inlet and outlet in the distribution plenum are also indicated. The direction of the incident x-ray beam with respect to the cooling channels was the same for the undulator and topography measurements; in both cases, the beam direction was along the cooling channels. Figure 3.b) indicates that, due to the convex shape, only a narrow strip in the central part of the crystal reflects at the top of the rocking curve. The origin of this curvature has to be investigated; it could be due in part to a flaw in the fabrication procedure and in part to bonding strains.

Since the die attach paste is composed mainly of glass and metal, we expect that the bond it makes will be resistant to radiation. The die attach paste bond is also compatible with water and liquid-nitrogen cooling[9] and is compatible with gallium for large bonding areas. Large-area bonds have maintained their strength for more than 2 years. On the other hand, because gallium dissolves the silver content of the paste and weakens the bond as it eats its way in, this paste is not appropriate for bonding the thin ribs of the heat exchanger of the gallium-cooled silicon crystals. In fact, the flexing of the heat exchanger under gallium pressure due to the weakening of the bond has been experimentally observed,[8] and the curvature shown in the topographic data could also be due, at least in part, to a degraded bond under the heat exchanger ribs.

If the strains induced by the bonding and fabrication processes could be eliminated, an option worth looking into would be the use of a glass-only paste, thus eliminating the gallium-incompatibility problem. We would need to find a supplier of a glass paste with a coefficient of thermal expansion similar to silicon over the temperature range of interest.

Si-Si and Si-Metal Epoxy Bonding

Another in-house bonding technique that we have used employs "green" epoxy.[10] This epoxy works well, from an adherence point of view, for both silicon-to-silicon and silicon-to-metal bonds. In the latter case, the metal has to have a coefficient of thermal expansion similar to silicon; we use a 39% Ni-Fe alloy[11] that matches the coefficient of thermal expansion of silicon from room temperature to 300 °C.[12] We have three crystals fabricated in this manner: a 4-inch-diameter (111) flat, a prototype 78°-inclined (111), and a prototype 85°-inclined (111).

The surfaces to be bonded are lapped and cleaned (soap and water, acetone, and alcohol). The epoxy is spread thinly on one of the mating surfaces, and the crystal is assembled and placed in the oven. A light weight (approximately 1-2 kg) is placed on top of the assembly. The temperature of the oven is slowly increased to 70 °C, roughly over a 4-hour period. The bond is left to cure for approximately 12-16 hours at this temperature. The oven is then turned off, and the parts are allowed to cool down inside the oven for approximately 24 hours.

This bond is compatible with gallium cooling, but the epoxy will probably degrade under radiation. The strain induced by the bonding is of the order of 2-4 arc seconds over an approximately 5-mm by 2-mm beam footprint,[8] as measured using the APS/CHESS prototype undulator on the slotted, flat crystal. A topographic study of the same crystal has shown an elaborate pattern of fabrication strains. Figure 4.a) is the rocking curve obtained when the x-ray illuminates the full face of the 4-inch-diameter crystal. The measured width is 15.5 arc seconds, which translates into a strain of 15.4 arc seconds. Figure 4.b) is the topograph taken at the top of the rocking curve, while Figure 4.c) indicates the direction of the incident x-ray beam with respect to the cooling slots. (For the undulator measurements, the x-ray beam was incident along the cooling channels.) The topograph in Figure 4.b) clearly shows the underlying cooling slots (recall that the topograph is the mirror image of the sample as seen by the beam). Because topographs were not taken at each stage of the fabrication process, it is difficult to assess the contributions of the different fabrication steps to the overall strain pattern, and/or whether part of the distortions are due to a weakening of the bond after being exposed to the undulator radiation.

Si-Si and Si-Metal Glass Frit Bonding

This bonding technique uses lead borosilicate glass frit as the bonding agent. Two different kinds of glass frit can be used, one to bond silicon to silicon, and the other for silicon to Ni-Fe bonds.[12] One restriction of the Si-metal bond is that it can only be performed over small areas (approximately 1 square cm); even a slight mismatch between the coefficients of thermal expansion can induce strains over a large-area Si-metal bond that may be severe enough to crack the silicon piece.

We have six cooled Si crystals that were fabricated using glass frit silicon-to-silicon bonding:[13]

1. a pin-post heat exchanger, flat (111), designed and fabricated by Rockwell International Corporation;[14]
2. a criss-cross heat exchanger, flat (111), with a tapered silicon distribution plenum;

3. a slotted heat exchanger, flat (111), 4" diameter crystal, with a tapered silicon distribution plenum;
4. a slotted heat exchanger, flat (111), 4" diameter crystal, with a 'standard design' plenum;
5. a variable-asymmetry (111) crystal, with a tapered silicon distribution plenum;
6. and a 90-mm by 120-mm, slotted heat exchanger, flat (111), prototype bend-magnet crystal.

Crystal #1 has four Ni-Fe metal coolant fittings bonded using the lower temperature glass frit; crystal #6 is bonded to a Ni-Fe manifold using a gold-based solder (see next section). All glass frit bonds were performed by Rockwell International Corporation.

The procedure is similar for both Si-Si and Si-metal bonds, but the kind of glass frit used is different. The surfaces to be bonded are lapped flat to better than 0.0005" and cleaned. Both surfaces are then sprayed with the glass frit, masking out, if necessary, any portions of the crystal (for example, the bottom of the heat exchanger channels). Then, each mating part is baked, and the parts are assembled. A weight is used to put some pressure on the bond (typically 2-3 psi), and the assembly is baked at 927 °C for the Si-Si bond and at 400 °C for the Si-metal bond.[12]

This type of bond satisfies the requirements of radiation resistance, compatibility with water and gallium cooling, and similarity of thermal expansion coefficient to that of silicon. In the case of the Si-Si bond, the strain measured over a few square millimeter area of the crystal is less than 2 arc seconds. We also conducted a detailed topographic study of crystal #3 listed above. Figures 5.a) and 5.b) show the rocking curves when the beam illuminates the full 4-inch-diameter and the central 1×1.4 cm^2 area of the crystal, respectively; the measured rocking curve widths are 3.5 arc seconds and 2.4 arc seconds, which indicate strains of 2.9 arc seconds and 1.3 arc seconds. Figure 5.c) is the topograph taken at the top of the rocking curve; the beam is incident along the direction of the cooling channels, see Figure 3.c). This crystal has not yet been tested at a synchrotron, but we expect that it will perform well, because it shows the least strains among the crystals that we have fabricated so far.

Similar topographic studies of the bend-magnet crystal (crystal #6 listed above) have shown strains of the order of 6 arc seconds over the entire crystal (90 mm by 120 mm).[15] Therefore, Si-Si glass frit bonding may not perform adequately for applications in which the footprint of the beam on the crystal is large.

Si-Metal Gold-Based Solder

This type of bond is appropriate for water cooling, but it may deteriorate if used with gallium, because gallium is known to attack gold. The surfaces to be bonded are prepared as for glass frit bonding: they are first lapped flat to within 0.0005", and then they are cleaned. The bonding process was carried out by Rockwell International Corporation;[14] since the process is proprietary, we do not know any details of the procedure.

The gold-based solder has been used in the fabrication of the 90-mm by 120-mm bend-magnet crystal (crystal #6 listed above). The first attempt at soldering the silicon assembly to the Ni-Fe was unsuccessful. The stainless steel inlet tubing and outlet tubing that were welded to the Ni-Fe manifold caused a local change of the coefficient of thermal expansion of the metal. This change was sufficient to result in large strains in the silicon-to-metal bond. Figure 6.a) is the rocking curve of the crystal when fully illuminated by the beam from the topography station. The broad base is due to the strain generated at the silicon-metal interface, while the sharper (but still wide) peak is an indication of the strains at the silicon-silicon bond (see previous section). Figure 6.b) is a superposition of several topographs taken at 4 arc seconds steps along the rocking curve. The wide stripe in the center corresponds to the reflection at the top of the rocking curve. The fringes at the sides are associated with the broad base on the rocking curve and show the strain field around the joints of the inlet and outlet stainless steel tubing to the Ni-Fe manifold. An attempt is currently underway to redo the silicon-to-metal bond. The silicon assembly has been sawed off the metal manifold, and the manifold has been redesigned to avoid generating a mismatch between the coefficients of thermal expansion of silicon and metal at the bonding interface.

No crystal with a soldered metal manifold has yet been tested by us at a synchrotron, so we have no experience on the performance of this bonding technique under operational conditions.

Conclusions and Future Research Plans

None of the techniques described above has (so far) yielded a strain-free bond, i.e., at the 1 arc second or less level, adequate for the fabrication of cooled single-crystal optics.

The direct bonding method works well for thin crystals, and we will carry out tests to determine if the technique can be extended to obtain a strain-free bond using thicker silicon pieces. We have procured the equipment that will allow us to develop the direct bonding procedures in-house: the spinner is operational, and an oven has been ordered. The chemical procedures required to treat the bonding surfaces have been established. We expect to be ready to try our hand at bonding thick silicon crystals in the near future.

Among the other techniques, silicon-to-silicon glass frit bonding has yielded some cooled crystals which are almost strain-free. Though the bond is still not perfect, this technique could provide a reasonable alternative if no other option is available.

Acknowledgments

The author is grateful to the members of the XFD-OP group for helpful discussions and for the information supplied for the compilation of this report. The topographic data was taken by Dr. Felix Krasnicki and Mr. Alan Philippides; their effort is gratefully acknowledged.

References

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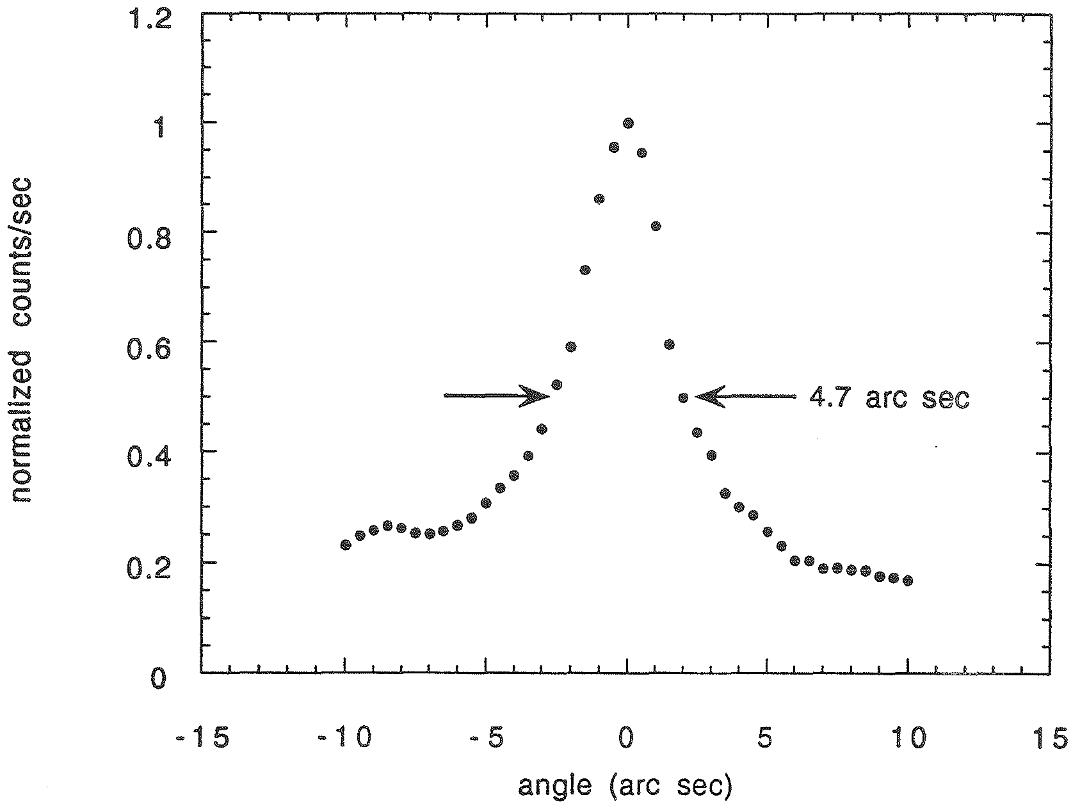
2. Kiyoshi Mitani and Ulrich M. Gösele, Journal of Electronic Materials, **21**, 669 (1992).
3. Bonded by Ulrich M. Gösele and collaborators at Duke University, Durham, North Carolina.
4. R.C. Blasdell, ANL, unpublished information, 1995.
5. Q.-Y. Tong, S. Krasnicki, and U.M. Gösele, Proceedings of the 3rd. International Symposium on Semiconductor Wafer Bonding, May 21-26, 1995, Reno, Nevada.
6. The die attach paste used is Johnson Matthey JM 4613 AuSub® Die Attach Paste.

7. The certificate of analysis provided by Johnson Matthey indicates that the die attach paste has 68% of silver (by weight), and a total of 85% of solid components (by weight); thus, organic components constitute 15% by weight.
8. D.M. Mills, W.K. Lee, R.K. Smither, and P.B. Fernandez, "High Heat Load Studies of Silicon and Diamond Monochromators Using the APS/CHESS Prototype Undulator," ANL/APS/LS-238, Argonne National Laboratory (1994).
9. R.K. Smither, ANL, unpublished information, 1995.
10. The epoxy used is 3M Scotch-weld 1838 B/A green epoxy.
11. 39% Ni, balance Fe alloy, specification ASTM B 753 T-39, supplied by Scientific Alloys, Inc., P.O. Box 523, Westerly, RI 02891.
12. Presentation by T. W. Tonnessen, Rockwell International Corporation, to XFD-OP staff on November 20, 1992.
13. A detailed description of the geometry of these silicon crystals can be obtained from R.K. Smither and W.K. Lee.
14. Rocketdyne-Albuquerque Operations, Rockwell International Corporation, 2511C Broadbent Parkway NE, Albuquerque, NM 87107.
15. S. Krasnicki, ANL, unpublished information, 1995. See also Figure 6.a).

a)



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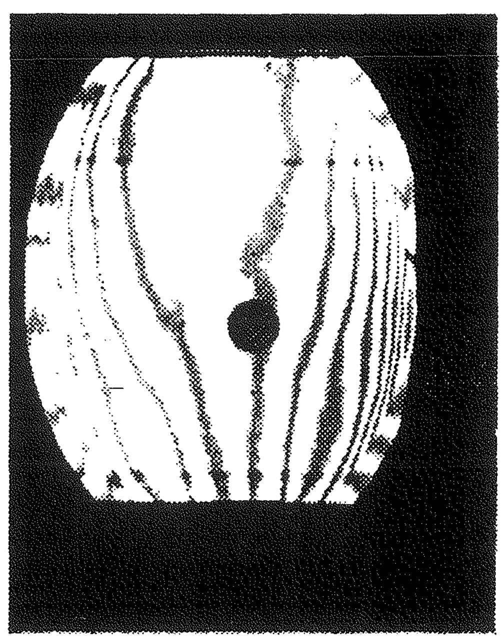


Figure 1 Data on the 4-inch-diameter, 2-mm-thick, direct bonded silicon wafers: a) (333) double-crystal rocking curve when the full face of the crystal is illuminated by the 8.05-keV x-ray beam from the topography unit; b) superposition of topographs taken at different points of the rocking curve, in 4 arc seconds steps.

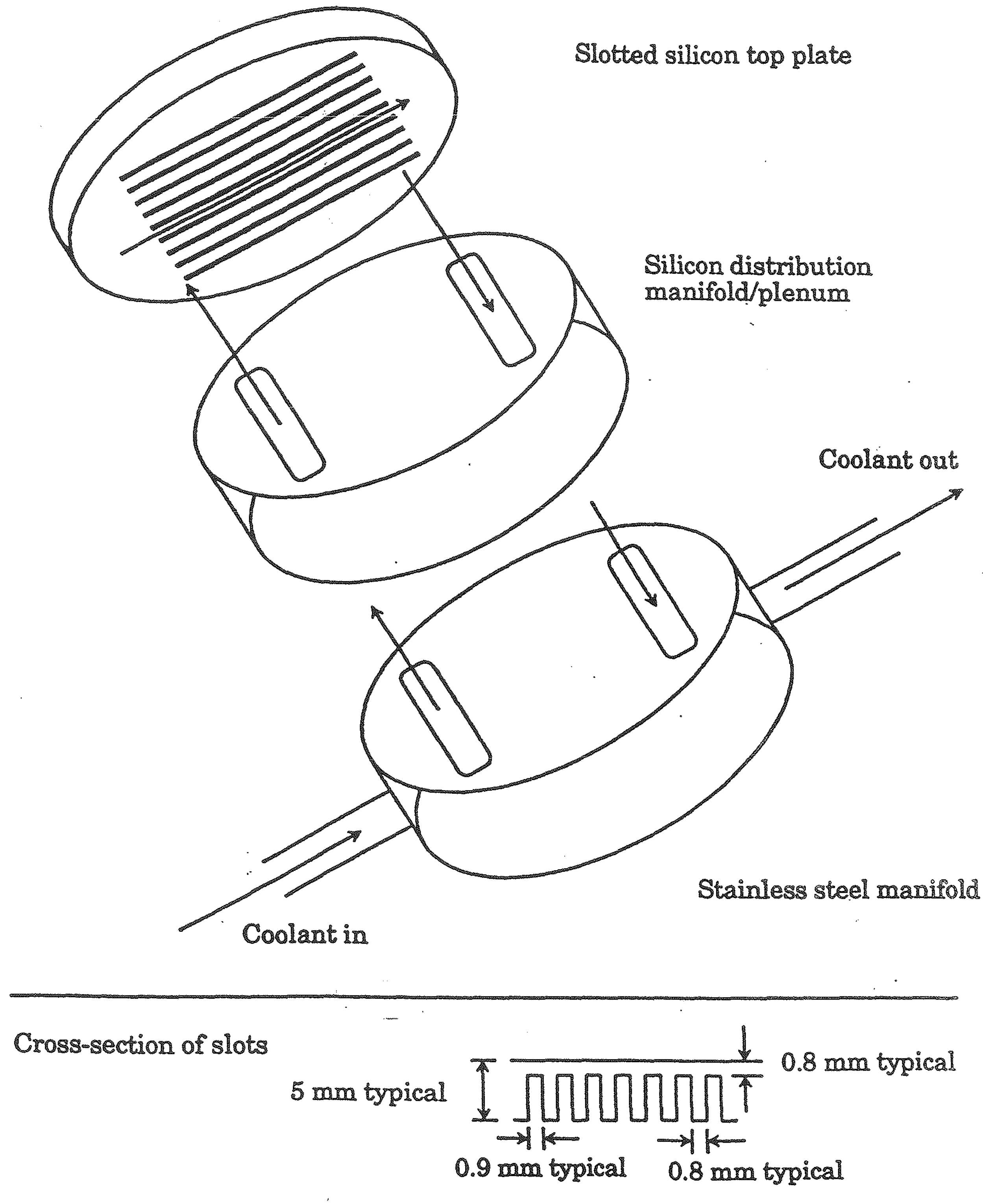
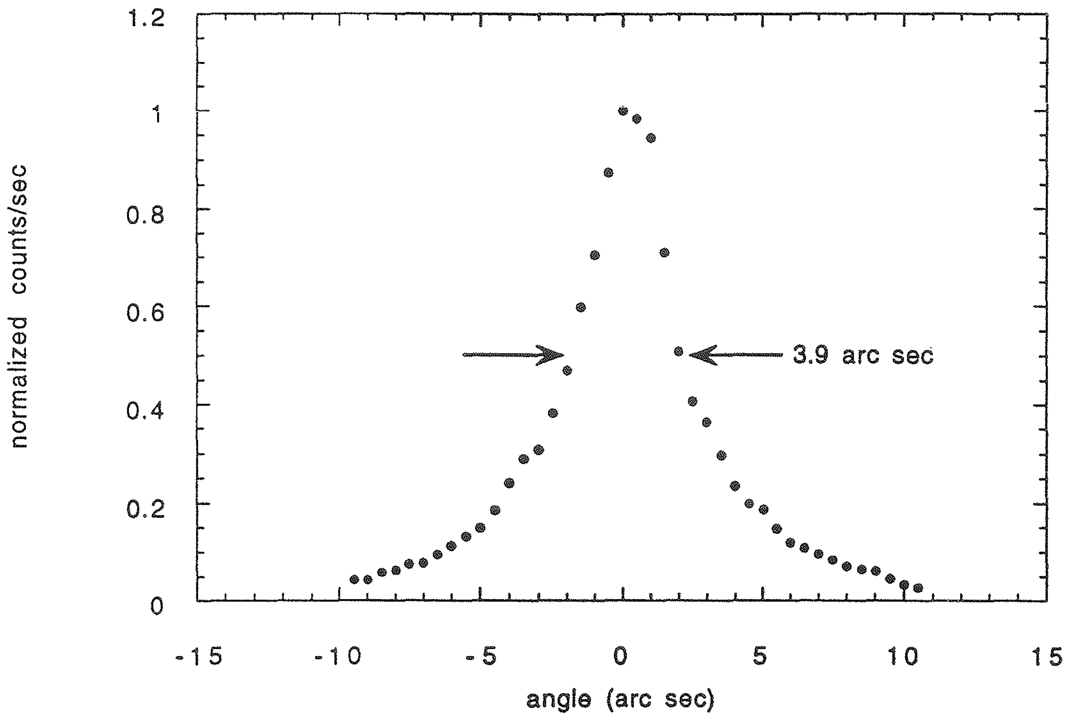


Figure 2 Sketch of a typical silicon-crystal assembly. The top plate is bonded to the distribution plenum, and the bonded crystal is then sealed to the metal manifold using rubber O-rings. (Figure courtesy of W.K. Lee.)

a)



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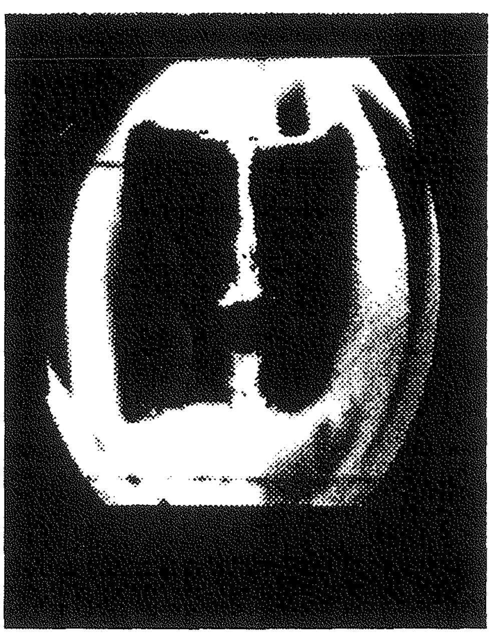


c)

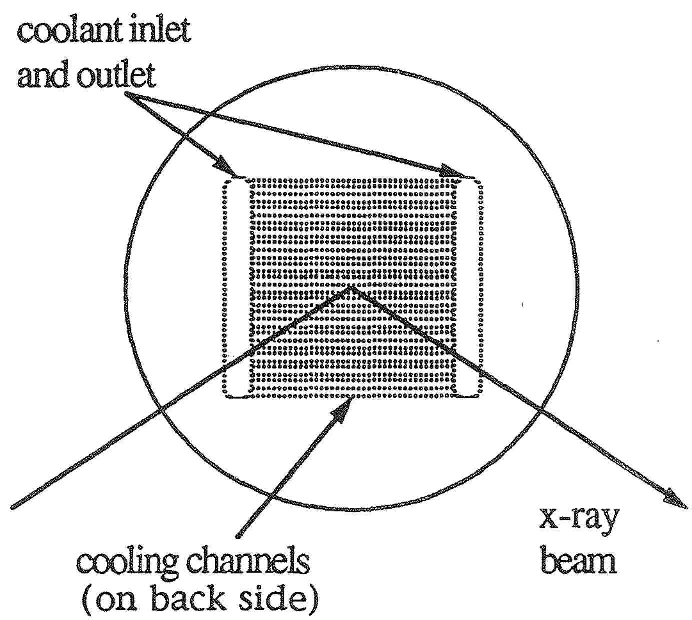


Figure 3 Topographic study of the 4-inch-diameter slotted, die attach paste bonded crystal: a) (333) double-crystal rocking curve obtained when the 8.05-keV x-ray beam illuminates the full face of the crystal; b) topograph taken at the top of the rocking curve; c) direction of the incident x-ray beam with respect to the cooling slots.

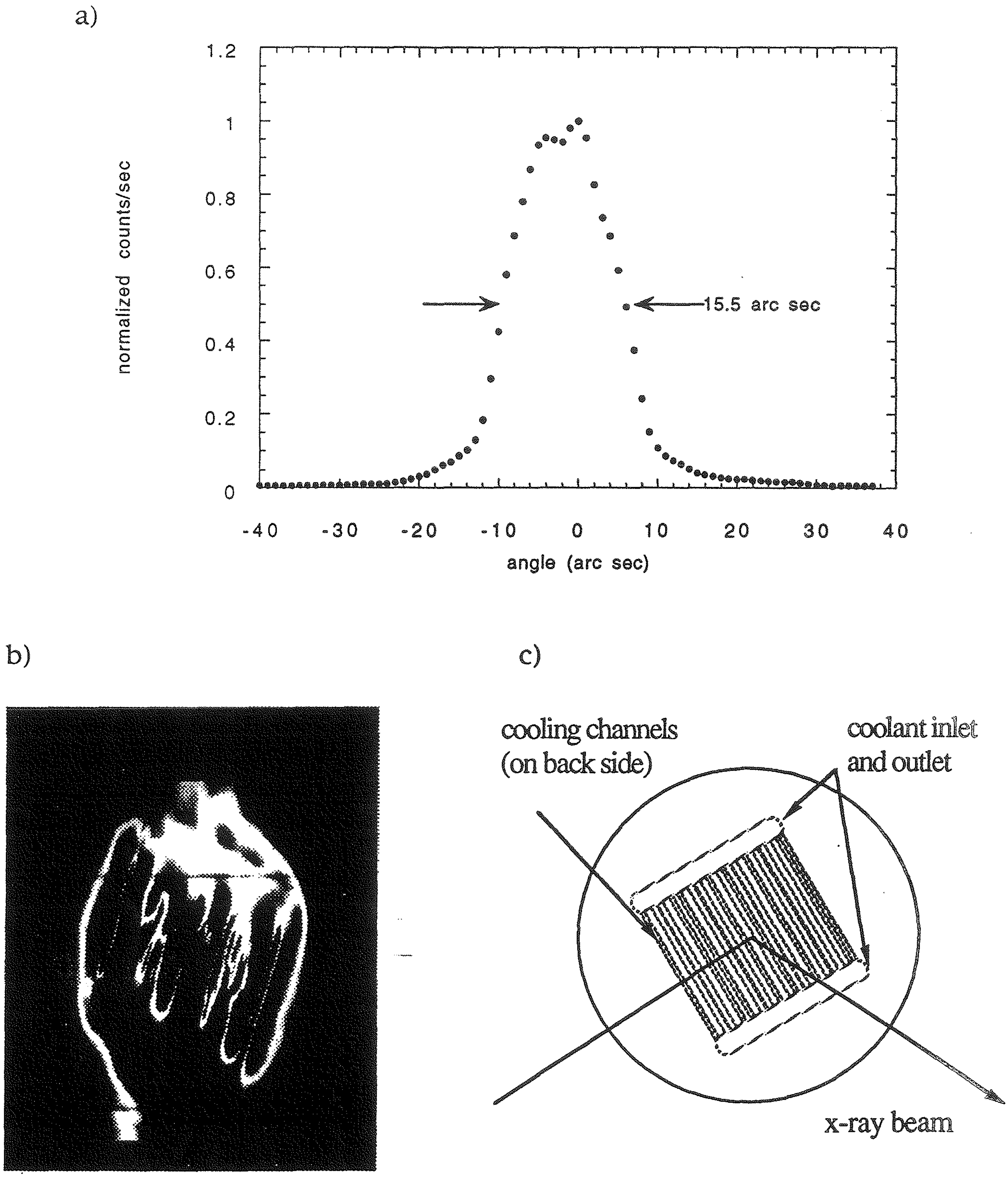
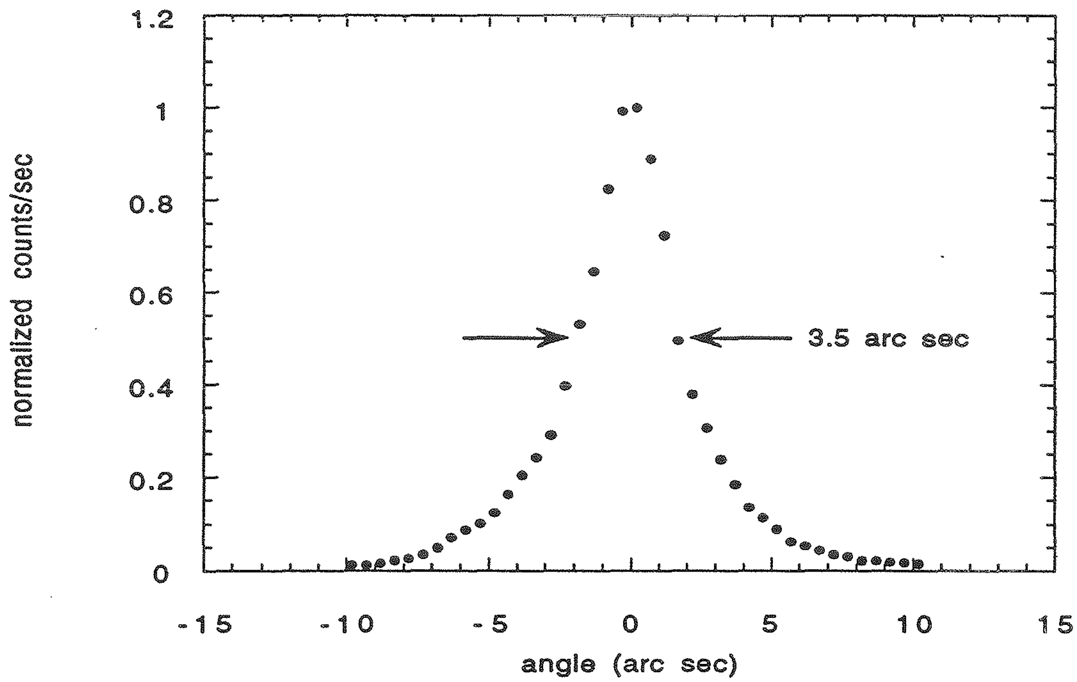


Figure 4 Topographic study of the 4-inch-diameter slotted, epoxy-bonded crystal: a) (333) double-crystal rocking curve obtained when the 8.05-keV x-ray beam illuminates the full face of the crystal; b) topograph taken at the top of the rocking curve; c) direction of the incident x-ray beam with respect to the cooling slots.

a)



1.2
1
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normalized counts/sec
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angle (arc sec)

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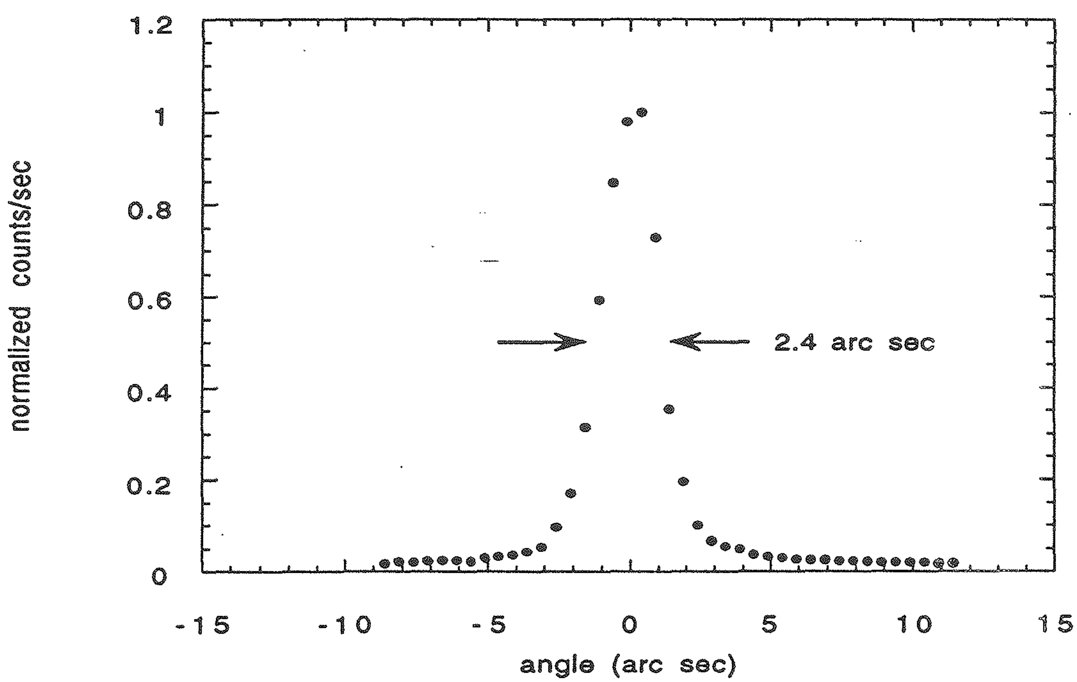

1.2
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angle (arc sec)

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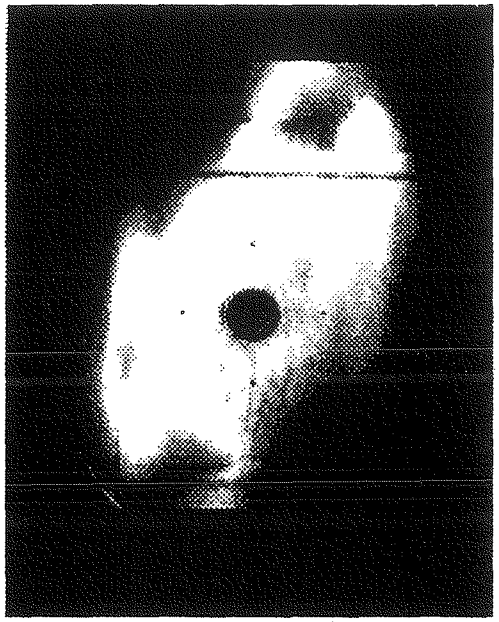
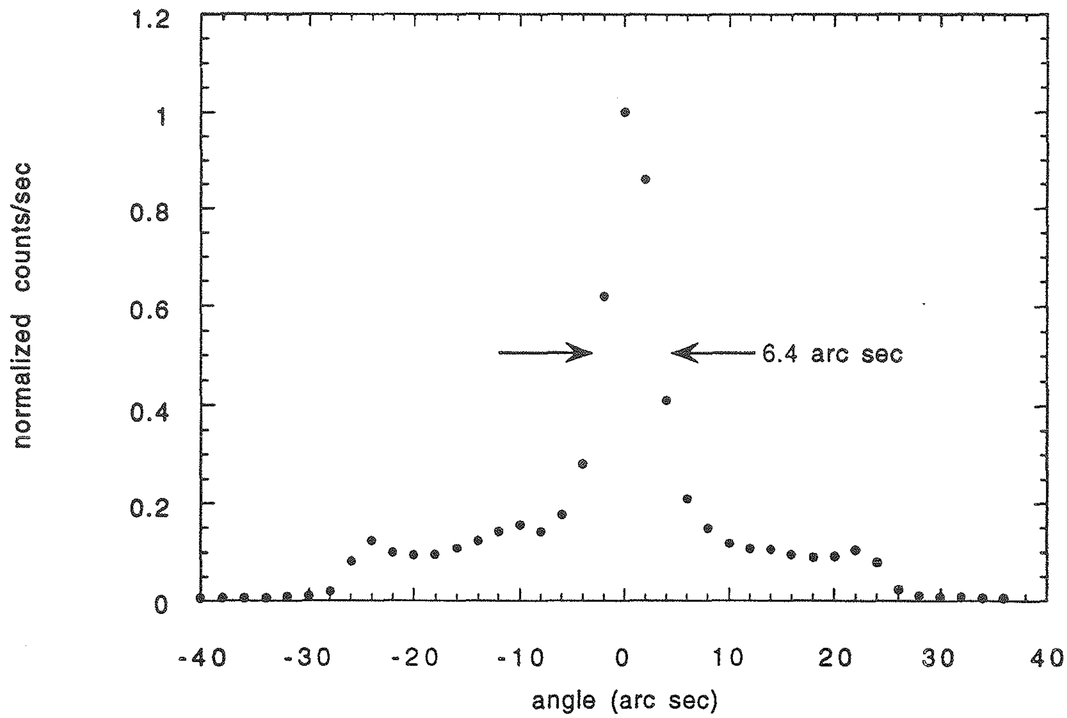


Figure 5 Data on the 4-inch-diameter slotted, frit-bonded crystal: a) and b) (333) double-crystal rocking curves when the 8.05-keV beam from the topography unit illuminates the full crystal and the central region (1×1.4 cm^2), respectively; c) topograph taken at the top of the rocking curve.

a)



b)

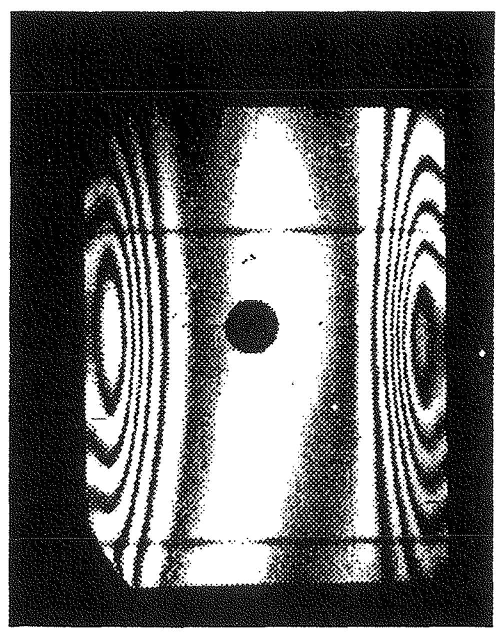


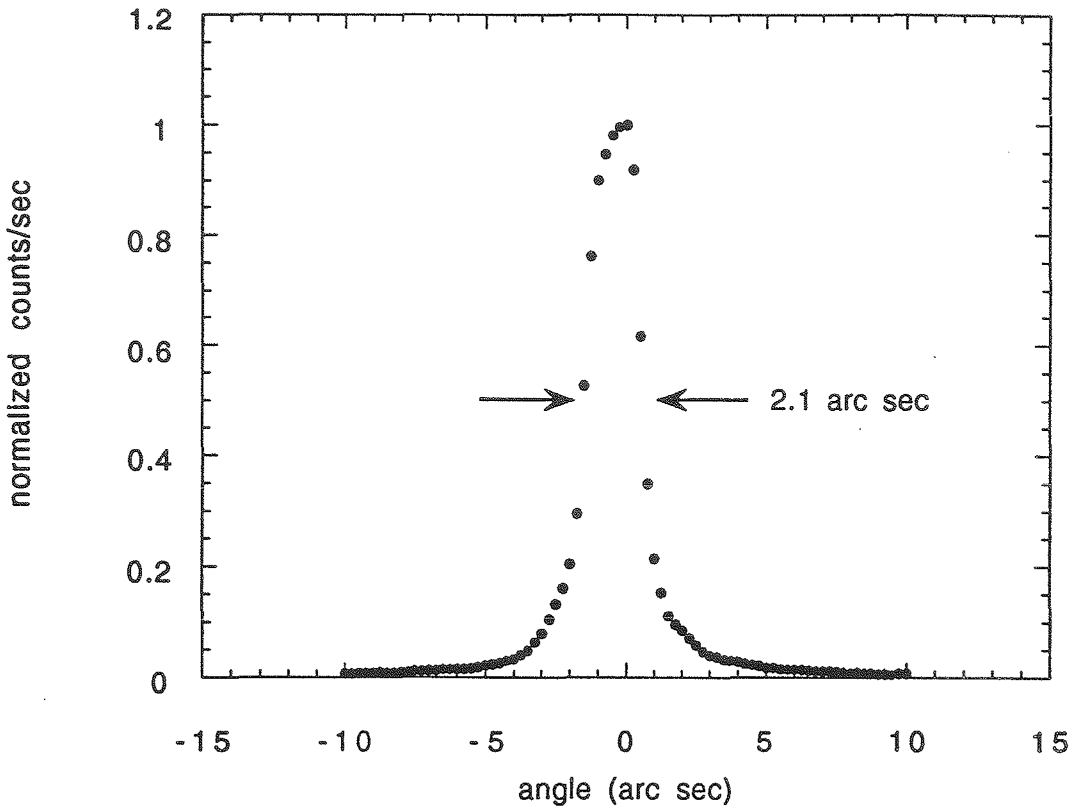
Figure 6 Data on the 90-mm by 120-mm bend-magnet crystal. The slotted heat exchanger is bonded using glass frit to the silicon distribution plenum, and the whole assembly is bonded to a metal manifold with a gold-based solder.
a) (333) double-crystal rocking curve when the 8.05-keV beam from the topography station illuminates a 90x90mm^2 area of the crystal; b) superposition of topographs taken at 4 arc seconds intervals along the rocking curve. The rings are due to localized strains from the silicon-to-metal bond.

Appendix A: Brief Description of the Topography Setup

In essence, the XFD-OP Topography Unit is a high-precision double-crystal diffractometer that utilizes an 18-kW rotating anode source and a broad, highly collimated monochromatic beam produced by an asymmetrically cut monochromator crystal. All the data described in this report were taken using a copper target and a silicon (111) monochromator crystal, with an asymmetric cut of 46.7°. The (333) reflection, with a Bragg angle of 47.5° was used. The shallow 0.8° incidence angle produced a 90 mm wide by 90 mm tall, highly collimated diffracted beam; only the Ka1 line was reflected. The size of the beam is determined by the monochromator size and by lead windows located after the monochromator crystal.

In this configuration, the double-crystal rocking curve from a perfect, symmetric (111) silicon second crystal reflecting in third order is expected to be 2.0 arc seconds. Figure A1.a) shows the rocking curve obtained with a strain-free 4-inch diameter, 10-mm thick (111) silicon wafer; the width of the (333) rocking curve is 2.1 arc seconds, in agreement with the theoretical "perfect" value. Figure A1.b) is the topograph taken at the top of the rocking curve; we see that for a perfect crystal, the whole face reflects at the same time. Note that the emulsion side of the photographic paper faces the crystal; the topograph is then a mirror image of the crystal as seen by the x-ray beam. Also note that the horizontal dimensions are contracted by a factor of 0.7 due to the 47.5° incidence angle on the second crystal.

a)



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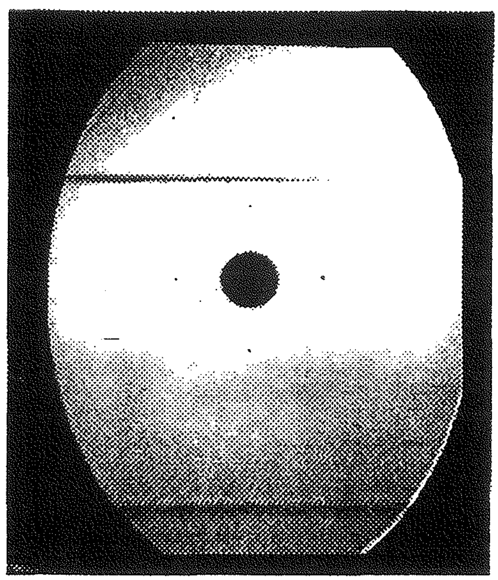

Figure A1 Topographic data on a strain-free 4-inch diameter, 10-mm thick (111) silicon wafer. a) (333) rocking curve when the 8.05-keV beam illuminates the full face of the crystal; b) topograph taken at the top of the rocking curve. The horizontal lines are shadows in the beam coming from the monochromator crystal.